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IS 1448-6 (1984): Methods of test for petroleum and its products, Part 6: Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter Method [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard
**METHODS OF TEST FOR
PETROLEUM AND ITS PRODUCTS**

[P : 6]

**HEAT OF COMBUSTION OF LIQUID HYDROCARBON FUELS
BY BOMB CALORIMETER METHOD**

(First Revision)

First Reprint DECEMBER 1998

UDC 662.767 : 536.626.2

*Adapted with permission from the Institute of Petroleum Standard No. IP 12/73
(Reapproved 1980)*

1. SCOPE

1.1 This method describes a procedure for the determination of the gross heat of combustion of liquid fuels and formulae are given for calculating the net heat of combustion.

2. SUMMARY OF METHOD

2.1 A weighed quantity of the sample is burned in oxygen in a bomb calorimeter under controlled conditions. The heat of combustion is calculated from the mass of the sample and the rise in temperature, with proper allowance for heat transfer and for the formation of nitric and sulphuric acids in the bomb, no account being taken of other minor products formed. The value so obtained is the gross heat of combustion at constant volume.

2.2 The bomb calorimeter is calibrated by the combustion of benzoic acid.

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2.3 Either *isothermal* or *adiabatic* calorimeter jackets may be used.

3. DEFINITIONS

3.1 Gross Heat of Combustion — The gross heat of combustion of a fuel at constant volume is the number of heat units measured as being liberated at 25°C when unit mass of the fuel is burned in oxygen saturated with water vapour in a bomb under standard conditions. The resultant materials in the bomb are considered as being gaseous oxygen, carbon dioxide, sulphur dioxide, nitrogen, liquid water in equilibrium with its vapour and saturated with carbon dioxide, other compounds in solution, and solid ash. The gross heat of combustion is represented by the symbol Q_g .

3.2 Net Heat of Combustion — The net heat of combustion of a fuel at constant pressure is the number of heat units measured as being liberated at 25°C when unit mass of fuel is burned in oxygen at constant pressure such that the heat released is equal to the gross heat of combustion of the fuel at constant pressure less than latent heat of evaporation at 25°C and constant pressure of the water both originally contained in the fuel and formed by its combustion. The net heat of combustion is represented by the symbol Q_n .

3.3 Unit of Measurement — The unit of measurement employed in this method is the joule. If the results are required in terms of calories per gram (Cal_{IT}/g) the following relationship may be used:

$$1 \text{ Cal}_{IT}/g = 4.1868 \text{ J/g (exactly)}$$

4. APPARATUS

4.1 The room in which the calorimeter equipment is located shall be free from draughts and sudden changes in temperature.

4.2 Calorimeter Bomb*

4.2.1 The bomb shall have an internal diameter of not less than 55 mm and a capacity of 325 ± 25 ml. The mass of the bomb shall not exceed 3.25 kg. It shall be so constructed that it does not leak, and can be easily drained. All parts enclosing the gas space shall be constructed of materials which are not affected sufficiently by the combustion process or products to introduce measurable heat input or alteration of the end products. The electrodes shall be made of heat and corrosion resistant material, for example, platinum or its alloys, suitable steels or nickel chrome, and so placed that when the oil cup is in position its base shall be not less than 90 mm from the underside of the bomb lid. The closure of the bomb shall be so designed that in normal operation the bomb can be sealed and opened by hand without the use of tools of any type. The calorimeter

*Detailed specification of one type of calorimeter bomb which meets the requirements of 4.2 are given in Annexure A of IP 12/73.

bomb seal shall be so designed that after hand-tightening an increase of pressure in the bomb will tighten the seal and prevent leakage

4.2.2 Pressure Test — After completion each bomb shall be tested under a water pressure of 300 bar, which shall be maintained in the bomb for a period of 10 minutes without leakage. After removal of the test pressure, the external diameter of the bomb body shall not show an increase exceeding 0.1 percent of the diameter measured, and the thread shall be examined for galling.

4.3 Calorimeter Vessel — Metal vessel polished on the outside, preferably at least 75 mm higher than the bomb, preferably of such cross-sectional area that 2 litres of water just covers the bomb.

4.3.1 Stirrer — A mechanical device for stirring the water in the calorimeter vessel at a uniform rate. The stirring shall be thorough but not so rapid as to cause appreciable heating of the water. Continuous stirring of the water in the calorimeter vessel of the isothermal jacketed apparatus for 10 minutes after thermal equilibrium has been established shall not cause an increase in temperature of more than 0.01°C. With the adiabatic jacketed apparatus continuous stirring of the water in the calorimeter vessel for 10 minutes after thermal equilibrium has been established, shall not cause an increase in temperature of more than 0.002°C. The portion of the stirrer immersed in the calorimeter vessel shall be connected to the outside portion with material of low heat conductivity.

4.3.2 Water Jacket — A double walled metal vessel containing water, fitted with a lid and leaving an air gap of preferably 10 mm all around the calorimetric vessel.

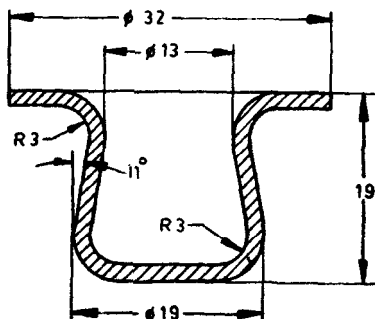
4.3.2.1 The isothermal jacket shall be arranged so as to remain at a uniform temperature (maximum variation 0.05°C) during the test.

4.3.2.2 Adiabatic calorimeters shall be capable of circulating water continuously through both jacket and lid. The system shall be fitted with heating and cooling devices such that the temperature of the jacket water is rapidly adjusted to that of the water in the calorimeter vessel with a sensitivity sufficiently good to satisfy the requirements of 9.5.2.

4.4 Fuel Cups — A range of fuel cups that may be used are illustrated in Fig. 1.

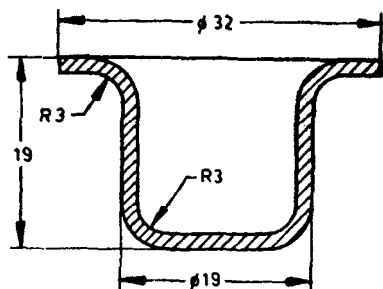
4.5 Ignition System — The ignition current should be drawn either from a stepdown transformer connected to an alternating current line or from a storage battery. The firing voltage shall not exceed 24 volts and should be held to the lowest practicable level recommended by the manufacturer. An ammeter or a pilot light may be used in the circuit to indicate when current is flowing, and the circuit shall be controlled by a double-pole, momentary contact switch which remains normally open,

except when held closed by the operator. The switch shall be held in the closed position for not more than two seconds.



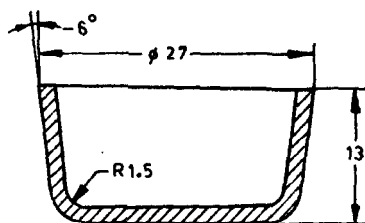
TYPE 1

MATERIAL	THICKNESS	APPROX MASS
Inconel	0.46	8.3 g



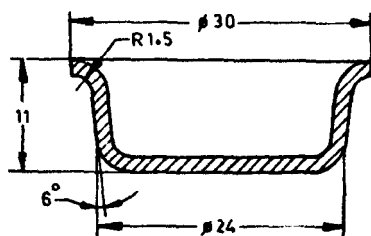
TYPE 2

MATERIAL	THICKNESS	APPROX MASS
Inconel	0.46	8.3 g



TYPE 3

MATERIAL	THICKNESS	APPROX MASS
Type 310 Stainless Steel	0.8	9 g
Hastalloy C	0.7	9 g
Platinum 3 1/2 percent Rh	0.35	11 g



TYPE 4

MATERIAL	THICKNESS	APPROX MASS
Platinum 3 1/2 percent Rh	0.35	11 g

All dimensions in millimetres.

FIG. 1 FUEL CUPS

4.6 Thermometers — A special mercury-in-glass calorimetric thermometer subdivided into graduations not greater than 0.01°C and covering a range 6°C ; a Beckmann thermometer with suitable subdivisions; or a thermoelectric thermometer of equal accuracy shall be used for determining the rise in temperature of the calorimeter water. The thermometer shall be certified by the National Physical Laboratory, New Delhi or any other recognized authority, to 0.005°C or preferably to 0.002°C and used with the corrections given on the certificate. Corrections shall be made for the temperature of the emergent stem of a mercury thermometer or for the setting of a Beckmann thermometer.

4.6.1 A mercury-in-glass calorimetric thermometer should be tapped lightly just before reading, to prevent errors caused by sticking of the mercury particularly when the temperature is falling. A convenient method of effecting this is to mount a small buzzer directly on the top of the thermometer, and to connect this to a dry cell and a push button; the button is pressed for a few seconds before each reading.

4.6.2 Thermometer Reading Eyepiece — Magnifying type with adjustable focus, for use with mercury-in-glass thermometers.

4.7 Oxygen Charging Equipment — The valves, gauges, filling tube and fittings used in the oxygen charging system shall meet industry safety codes and shall be rated for use at input pressures up to 206 bar and discharge pressures up to 55 bar. Separate gauges should be provided to show the supply pressure and the bomb pressure. The bomb gauge should be not less than 75 mm in diameter and preferably graduated from 0 to 55 bar in 1 bar sub-divisions. Both gauges shall be absolutely oil-free and shall never be tested in a hydraulic system containing oil. The charging equipment should include either a pressure reducing valve which will limit the discharge pressure to a maximum of 40 bar or a relief valve set to discharge at 40 bar in case the bomb should accidentally be overcharged. Means should also be provided for releasing the residual pressure in the filling tube after the bomb valve has been closed.

5. MATERIALS AND REAGENTS

5.1 Oxygen — Under pressure in cylinders, free from combustible material.

5.2 Ignition Wire — 0.1 ± 0.03 mm diameter platinum wire. The same diameter and length of wire should be used in the calibration and determination.

5.3 Firing Cotton (if Required) — A strand of wick as specified for use with IS : 1448 [P : 34]*, dried at 100 to 105°C and stored over silica

*Determination of sulphur in petroleum products (lamp method) (second revision)

gel until required. The heat of combustion of each batch of firing cotton shall be determined.

5.4 Sodium Hydroxide Solution — Approximately 0.1 N, accurately standardized.

5.5 Benzoic Acid — Powder or pellet form of thermochemical standard of purity and certified heat of combustion. Powdered materials shall be compressed into pellets.

5.6 Cellulose Adhesive Tape — Plain-transparent cellulose film of width not less than 25 mm and thickness 0.05 to 0.075 mm. Use only cellulose-based film. The heat of combustion of each batch of tape shall be determined.

5.7 Indicator — The following alternatives may be used.

5.7.1 Methyl Purple Solution — Aqueous solution containing approximately 0.1 percent active constituent.

5.7.2 Methyl Red Solution — 60 percent alcohol-aqueous solution containing approximately 0.1 percent by mass indicator.

6. SAFETY PRECAUTIONS

6.1 The mass of sample shall be such that the heat developed during the combustion shall in no case exceed 90 J/ml bomb capacity; and the total mass of sample, together with any combustible material such as cellulose tape, shall be calculated in advance, so that the given limit is not exceeded.

6.2 Provision shall be made to ensure that the oxygen filling pressure does not exceed the safety limit (*see 4.7*).

6.2.1 The operator shall not go near the calorimeter until at least 20 seconds have elapsed from the time of firing.

6.3 The bomb shall be completely submerged in water during firing and the charge shall not be ignited if there is any evidence of gas leakage. Also, do not fire the bomb if it has been accidentally over-charged with oxygen to a pressure greater than 40 bar.

6.4 Because of the risk of explosion, it is strongly recommended that the firing point and the oxygen cylinder be situated outside the room or safety enclosure containing the calorimeter, but in any case firing shall be by remote control in order to minimize the risk of injury to the operator (*see 4.5*).

7. SAMPLE

7.1 Take samples of liquid fuels of flash point less than 38°C directly into a container of not less than 200 ml capacity, fill the container as completely as possible and close immediately after taking the sample. Store samples in a refrigerator at below 4°C until required. The fuel shall be transferred to the fuel cup at a temperature as near as possible to the storage temperature.

7.2 If results are required on a dry basis, determine the water content independently and correct the result accordingly.

8. STANDARDIZATION

8.1 Determination of Effective Heat Capacity Energy Equivalent or Water Equivalent — Using tweezers, place in the weighed fuel cup (Types 2 or 3 of Fig. 2) the approximate mass of benzoic acid derived from the relation:

$$m = \frac{90 \times \text{Bomb capacity in ml}}{26\,500}$$

where

m = approximate mass of benzoic acid in g.

8.1.1 Weigh the fuel cup and its contents to the nearest 0.1 mg to find the mass of benzoic acid. Pipette 1 ml of water into the bomb (to saturate the oxygen used for combustion with water vapour). Place the fuel cup in the circular ring support attached to the terminals of the bomb.

8.1.1.1 Attach a measured length of ignition wire (approx 100 mm, see 5.2) across the bomb terminals so as to form a U-shape loop and arranged so that the central portion of the loop is in contact with the benzoic acid.

OR

8.1.1.2 Attach a measured length of ignition wire (approx 100 mm, see 5.2) across the bomb terminals under light tension. Weigh a suitable length of dry firing cotton and tie it at the centre of the ignition wire. Insert the free end of the firing cotton into the crucible so that it is in contact with the benzoic acid.

8.1.2 Close the bomb securely.

8.1.3 Admit oxygen slowly to a gauge pressure of 30 bar.

8.1.4 Determine the effective heat capacity of the bomb by making not less than 5 combustions of the certified benzoic acid using the standard

quantity of water. The standard quantity of water is such that the cover of the bomb (but not terminals) is immersed and the effective heat capacity of the system is approximately 10 500 joules/°C. If necessary make preliminary combustions of benzoic acid to establish the correct mass of water. Once established this shall be the standard quantity of water, weighed to within 0.5 g, to be used in all subsequent determinations.

8.1.5 When using *isothermal* apparatus follow the details of procedure described in 9.4 and 9.6. When using *adiabatic* apparatus follow the details of procedure described in 9.5 and 9.6. Calculate the effective heat capacity as described in 10.1 to 10.4.

9. PROCEDURE

9.1 For liquid fuels of flash point below 38°C proceed as in 9.1.2 or 9.1.3 and continue the determination as in 9.4, 9.6 and 9.7 when using *isothermal* apparatus or as in 9.5, 9.6 and 9.7 when using *adiabatic* apparatus. For liquid fuels of flash point above 38°C proceed as in 9.2.1 or 9.2.2 and continue the determination as in 9.4, 9.6 and 9.7 when using *isothermal* apparatus or as in 9.5, 9.6 and 9.7 when using *adiabatic* apparatus.

9.1.1 *Liquid Fuels of Flash Point Less than 38°C*

9.1.2 Fuel Cup Type 1 or 4 of Fig. 1 — Weigh the fuel cup to nearest 0.1 mg. Cover the cup with the cellulose adhesive tape, trim around the edge with a razor blade, and seal tightly. Puncture the tape with a large needle to make an opening somewhat larger than the diameter of the hypodermic syringe needle. Place a 4 × 12 mm strip of tape, creased in the middle and sealed along one side, in the centre of the tape, disk to give a flap arrangement which will cover the hole when finally sealed down, as shown in Fig. 2, Type 4. Weigh the cup and tape, and remove from the balance with forceps. Add the required mass (*see* Note under 9.1.3) of sample with a hypodermic syringe through the hole in the tape. Seal the flap down over the hole by pressing lightly with a metal spatula. Allow the temperature to reach equilibrium (*see* 7.1) and reweigh the cup, tape and sample. Care shall be taken throughout the weighing and filling operation to avoid contacting the tape or cup with bare fingers. Place the cup in the curved electrode and attach a measured length of ignition wire (*see* 5.2) across the bomb terminals such that the central portion of the loop presses down on the centre of the tape disk. Pipette 1 ml of water into the bomb and immediately close the bomb securely.

OR Alternatively

9.1.3 Fuel Cup Type 1 or 4 of Fig. 1 — Weigh the fuel cup to the nearest 0.1 mg. Cover the fuel cup top with two pieces of cellulose adhesive tape, such that they slightly overlap as shown in Fig. 2, Type 1. Fix one end of a weighed length of firing cotton into the overlap, the other end being free

for attachment to the ignition wire. Weigh the fuel cup and the attached tape and firing cotton to the nearest 0.1 mg. Charge the fuel cup by lifting the lip of the sealing tape and introducing the required mass (*see Note*) of liquid fuel with a hypodermic syringe. Replace the tape to seal the fuel cup, allow the temperature to reach equilibrium (*see 7.1*) and weigh the fuel cup and its contents to the nearest 0.1 mg. Pipette 1 ml of water into the bomb. Place the fuel cup in the circular ring support attached to the terminals of the bomb. Attach a measured length of ignition wire (*see 5.3*) across the bomb terminals under light tension. Tie the free end of the firing cotton to the centre of the ignition wire and with a pin make about six small holes in the tape sealing the mouth of the fuel cup. Immediately close the bomb securely.

NOTE — The mass of the sample shall be controlled such that

$$M = \frac{90 \times \text{Bomb capacity in ml}}{Q_g}$$

where

M = required mass of sample, in g, and

Q_g = approx heat of combustion of the sample in J/g.

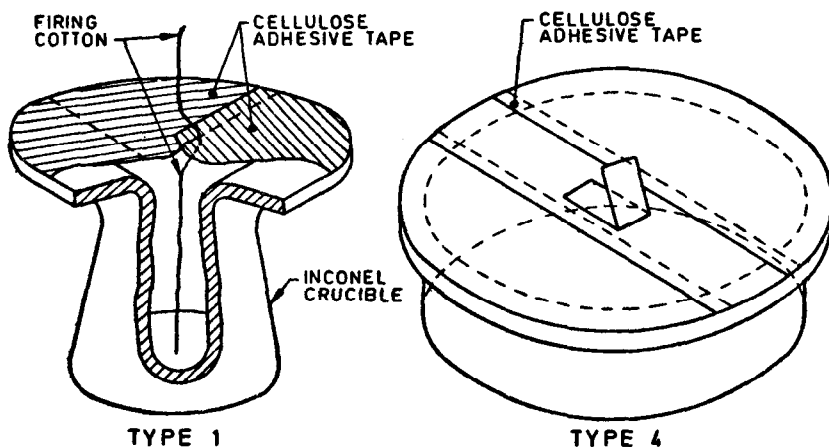


FIG. 2 SEALED FUEL CUP ASSEMBLIES

9.2 Liquid Fuels of Flash Point Greater than 38°C — (*see Note under 9.2.2*).

9.2.1 Fuel Cup Type 2, 3 or 4 of Fig. 1 — Place the required mass (*see* Note under **9.1.3**) of liquid fuel in the weighed fuel cup. Weigh the fuel cup and its contents to the nearest 0.1 mg. Pipette 1 ml of water into the bomb. Place the fuel cup in the circular ring support attached to the terminals of the bomb. Attach a length of ignition wire across the bomb terminals under light tension. Weigh a suitable length of dry firing cotton and tie it at the centre of the ignition wire. Dip the free end of the firing cotton into the contents of the fuel cup and immediately close the bomb securely.

OR Alternatively

9.2.2 Fuel Cup Type 2, 3 or 4 of Fig. 1 — Place the required mass (*see* Note under **9.1.3**) of sample in the weighed fuel cup. Weigh the cup and sample to nearest 0.1 mg. Place the cup in the curved electrode and arrange the fuse wire so that the central portion of the loop just dips into the contents of the cup. Pipette 1 ml of water into the bomb. Immediately close the bomb securely.

NOTE — Liquid fuels having flash point in the range 38 to 60°C may tend to lose a small proportion of their more volatile components on exposure to the atmosphere. Weigh the sample into the fuel cup and seal immediately in the bomb whilst avoiding exposure to draughts.

9.3 Admit oxygen slowly to a gauge pressure of 30 bar.

9.4 Isothermal Jacket Calorimeters — Fill the water jacket with water at a temperature within 3°C of room temperature and ensure that it remains constant throughout. Fill the calorimeter vessel with the standard quantity of water (*see* **8.1.5**) weighed to within 0.5 g. The initial temperature of the bomb and calorimeter shall be:

- i) in the range 15 to 32°C;
- ii) above the dew point;
- iii) approx 2.5°C below the temperature of the water jacket and preferably within 2°C of the temperature of standardization (*see* Note under **10.4**).

9.4.1 Place the prepared bomb, **CAUTION** (*see* **6**) in the calorimeter vessel, check that there is no leakage of oxygen, confirm that the firing leads are dead, and make the appropriate electrical connections. Put the covers in position, arrange the thermometer and stirrer so that they do not touch the bomb or vessel, and start the stirrer.

9.4.2 When the temperature is rising steadily and at least 5 minutes after starting the stirrer, record a series of temperature readings (*see* Note 1) at 1 minute intervals for 5 minutes from which the temperature at the end of the sixth minute that is, time of firing will later be found by

extrapolation. The rate of temperature-rise in the 5 minutes period before firing shall be constant (*see* Note 2).

NOTE 1 — Temperatures should be read to 0.001°C. This will not be possible during the rapid period, and the most accurate readings which are practicable at this time should be recorded. Mercury-in-glass thermometers shall be tapped lightly before reading, particularly when the temperature is falling, to prevent errors caused by sticking of the mercury. A convenient method of effecting this is to mount a small buzzer directly on top of the thermometer, and operate it for a few moments before each reading. Use an eyepiece to facilitate thermometer reading.

NOTE 2 — The rate of temperature change with time shall be considered constant if the variation in the rate does not exceed 0.001°C/min. The rate of change of temperature shall be calculated as the mean of the relevant recordings.

9.4.3 At the end of the sixth minute fire the charge by closing the firing circuit for not longer than 2 seconds.

CAUTION (*see* 6.2) : Do not go near the bomb until at least 20 seconds have elapsed from the time of firing.

9.4.4 Record the temperatures at 45, 60, 75, 90 and 105 seconds after the time of firing. By interpolation between these readings the time at which the temperature-rise reached 60 percent of its final value will later be found (*see* Note).

NOTE — When the approximate heat of combustion of the fuel is known to within 1 500 joule/g, the expected temperature-rise may be determined from the relation:

$$t = \frac{MQ_g}{W}$$

where

Q_g = approx gross heat of combustion, in J/g;

M = mass in g of sample; and

W = effective heat capacity of calorimeter in J/°C.

9.4.5 After the rapid rise period (about 4 to 5 min) record the temperature at 1 minute intervals on each completed minute after the time of firing. Continue these readings until the difference between successive readings has been constant for 5 minutes (*see* Note under 9.4.4). From this series determine the time at the beginning of the period in which the rate of temperature change with time is constant. Determine also the rate of change of temperature with time.

9.4.6 The temperature rise shall be between 2.5°C and 3.5°C and the final temperature shall not be more than 1°C (and preferably not more than 0.5°C) above that of the jacket.

Calculate the temperature at the 60 percent point by adding to the temperature at the time of firing, 60 percent of the expected temperature-rise. Start an additional stopwatch at the time of the first reading of the series of temperature recordings made before firing. Stop the stopwatch

when the calculated 60 percent temperature-rise is attained and record the time.

9.5 Adiabatic Calorimeters — Fill the calorimeter vessel with the standard quantity of water, weighed to within 0.5 g (*see* 8.1.5), the temperature of which shall be within the range of 15 to 32°C, shall be above the dew point and preferably within 2°C of the temperature of standardization (*see* Note 2 under 10.4). Ensure that the jacket is filled with water at a temperature close to, and preferably equal to or slightly below that of the calorimeter vessel. Place the prepared bomb in the calorimeter vessel, check that there is no leakage of oxygen, confirm that the firing leads are dead and make the appropriate electrical connections. Place the covers in position, arrange the stirrer, thermometer, and temperature regulating device in position so that they do not touch the bomb or calorimeter walls. Switch on the calorimeter stirrer, jacket stirrer, heaters, and cooling coil and run the apparatus for 5 minutes to attain equilibrium.

9.5.1 If, at the start, the bomb is markedly different in temperature from the calorimeter vessel more than 5 minutes may be required to reach equilibrium.

9.5.2 Check that the apparatus is at equilibrium by recording the temperature to the nearest 0.001°C (*see* Note 1 under 9.4.2) at 1 minute intervals. Successive readings shall not differ by more than 0.001°C and shall not show any progressive tendency. When four successive readings have complied with the requirements consider the apparatus to be at equilibrium and record the temperature to the nearest 0.001°C as the firing temperature (t_a , *see* 10.3). Fire the charge by closing the firing circuit for not longer than 2 seconds.

CAUTION (*see* 6.2) : Do not go near the bomb until at least 20 seconds have elapsed from the time of firing.

9.5.3 Record the temperature to the nearest 0.001°C at 1 minute intervals until the same temperature is recorded for three successive readings. Record this value as the final temperature t (*see* 10.3) (*see* Note).

NOTE — If a constant temperature is not obtained within 15 minutes of firing the apparatus should be considered suspect.

9.6 Fifteen minutes after the ignition, remove the bomb from the calorimeter, dry the outside of the bomb and release the pressure uniformly at such a rate that the operation takes at least 1 minute. Open the bomb and examine the inside for traces of unburned oil or soot; if either is found, discard the determination (*see* Note).

NOTE — The combustion of highly leaded fuels may cause appreciable corrosion inside a stainless steel bomb, but this is not to be taken as reason for rejecting the test result.

9.6.1 Rinse out the interior of the bomb, including the fuel cup, with a fine jet of distilled water, and collect the washings, which shall not exceed a volume of 350 ml, in a beaker. Add a few drops of indicator and titrate with the sodium hydroxide solution to determine the quantity of nitric and sulphuric acids formed in the combustion.

9.7 When the sulphur content of the sample is unknown, filter the neutralized washings through a medium filter paper such as a Whatman No. 40, and determine the sulphur content as prescribed in IS : 1448 [P : 33]*:

10. CALCULATION AND REPORT

10.1 Correct all temperatures for thermometer errors according to the certificate.

10.2 Temperature-Rise in Isothermal Jacket Calorimeter — Using data obtained as prescribed in 9.4, compute the temperature-rise, Δt , in an isothermal jacket calorimeter as follows:

$$\Delta t = t_0 - t_a - r_1 (b - a) - r_2 (c - b)$$

where

Δt = corrected temperature-rise, in °C;

a = time for firing, in minute;

b = time (to nearest 0.1 minutes) when the temperature-rise reaches 60 percent of the total, in minute;

c = time at beginning of period in which the rate of temperature change with time has become constant (after combustion), in minutes;

t_a = temperature at time of firing, in °C;

t_0 = temperature at time, c , in °C;

r_1 = rate at which the temperature was rising during the 6 minutes period before firing, in °C/min;

r_2 = rate at which the temperature was rising during the 5 minutes period after time c in °C/min. If the temperature was falling r_2 is negative and the quantity [$- r_2 (c - b)$] is positive.

*Methods of test for petroloum and its products : [P : 33] Sulphur by bomb method.

10.3 Temperature-Rise in Adiabatic Jacket Calorimeter — Using data obtained as prescribed in 9.5 and 10.1 compute the temperature-rise, Δt in an adiabatic jacket calorimeter as follows:

$$\Delta t = t_f - t_a$$

where

Δt = corrected temperature-rise, in °C;

t_a = temperature at time of firing, in °C; and

t_f = final maximum recorded temperature, in °C.

10.4 Effective Heat Capacity of the Calorimeter — Calculate the effective heat capacity of the calorimeter, W in joules/°C as follows:

$$W = \frac{[Q_B + 1.2 (25 - t_f)] m + e_1 + e_2}{\Delta t} - D_T \text{ (See Notes 1 and 2)}$$

where

Q_B = certified calorific value of the benzoic acid in J/g (see 5.5);

m = mass of benzoic acid, in g;

Δt = corrected temperature-rise, in °C;

e_1 = correction for the heat of formation of acids in J (1 ml 0.1 N NaOH = 5.9 J);

e_2 = correction for heat of combustion of firing cotton in J (if used) (heat of combustion of batch $\times$ mass of cotton); and

D_T = correction for test temperature variation (see Note 2) in J/°C. (apply this correction in whole numbers).

NOTE 1 — The term $[Q_B + 1.2 (25 - t_f)]$ relates the certified of combustion of benzoic acid at 25°C to the final temperature of the calorimeter.

NOTE 2 — The term D_T is an approximation which is applied to adjust the effective heat capacity of the system at the mean temperature, T of the determination to the reference temperature of 25°C, and is only necessary when the mean temperature of the bomb standardization and the determination differ by more than 3°C. It is calculated as follows:

$$D_T = \alpha M_W - \beta \left(M_S + \frac{M_B}{4} \right) \text{ J/deg C.}$$

where

M_W = mass of water in the system, in kg;

M_S = mass of stainless steel in the system in kg; and

M_B = mass of bronze and brass in the system in kg.

The values α and β are obtained from the graph in Fig. 3. The mean temperature T in $^{\circ}\text{C}$ is given by the relation:

$$T = \frac{t_a + t_f}{2}$$

where

t_a = temperature at the time of firing in $^{\circ}\text{C}$; and

t_f = final maximum recorded temperature in $^{\circ}\text{C}$.

Plot the calculated values of D_T in $\text{J}/^{\circ}\text{C}$ against values of T between 15 and 35°C .

10.5 Gross Heat of Combustion — Calculate the gross heat of combustion at constant volume and at 25°C in J/g from the equation:

$$Q_g = \frac{\Delta t \times (W + D_T) - e_1 - e_2 - e_3 - e_4}{M}$$

where

W = effective heat capacity of the system at 25°C , in $\text{J}/^{\circ}\text{C}$;

D_T = correction at the mean temperature, T obtained from the data plotted as described in Note 2 under 10.4, in $\text{J}/^{\circ}\text{C}$ (apply this correction in whole numbers);

Δt = corrected temperature-rise, in $^{\circ}\text{C}$;

e_1 = correction for the heat of formation of acids, in joules (1 ml 0.1 N NaOH = 5.9 J);

e_2 = correction for firing cotton (if used) in joules (heat of combustion of batch $\times$ mass of cotton used);

e_3 = is a supplementary correction for the sulphur content of the sample, in joules ($58.6 \times$ percent sulphur $\times$ mass of sample, in g);

e_4 = correction for cellulose tape, if used, in joules (heat of combustion of batch $\times$ mass of tape used); and

M = mass of sample, in g.

10.6 Net Heat of Combustion — Calculate the net heat of combustion (Q_n) in joules/g from the following equation:

$$Q_n = Q_g - 212.19H$$

where

H = is the hydrogen content of the sample in percent by mass.

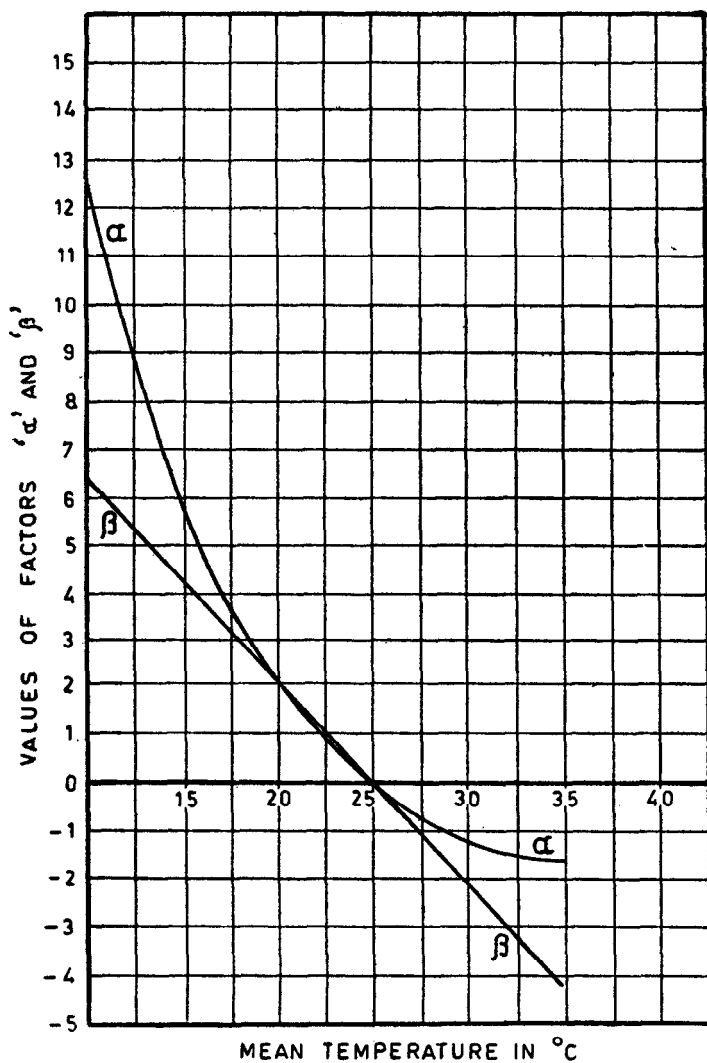


FIG. 3 VALUES OF α AND β FOR CALCULATING D_T

10.6.1 The following equation may be used for the calculation of net heat of combustion of gasoline and turbine fuel when the hydrogen content of the sample is not known:

$$Q_n = 10\,023 + 0.7195 Q_g \text{ (see Note)}.$$

NOTE — This equation is only recommended if the percentage of hydrogen is not known. It is based on the previous equation and an empirical relationship between Q_n and the percentage of hydrogen in aviation gasoline and turbine fuels, developed from data by R. S. Jessup and C. S. Gragoe, 'Heat of Combustion of AN-F-28 Aviation Gasoline', National Advisory Committee of Aeronautics, Tech. Note 996, June 1945, and J. A. Coglianò and R. S. Jessup, 'Relationship between Net Heat of Combustion and Aniline Gravity Product of Aircraft Fuels, National Bureau of Standards Report 2348, March 1953.

10.7 To obtain the gross or net heat of combustion in cal_{IT}/g divide Q_g or Q_n by the factor given in 3.

10.8 Report the result as the Gross or Net Heat of Combustion to the nearest 20 J/g or to the nearest 5 cal_{IT}/g.

11. PRECISION

11.1 Repeatability — Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

$$276 \text{ J/g (66 cal}_{IT}\text{/g)}$$

11.2 Reproducibility — The result submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amount:

$$773 \text{ J/g (185 cal}_{IT}\text{/g)}$$